FCS01- SOP for Detecting Controlled Dangerous Substances

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1. Scope

1.1. This method outlines the analytical procedure for the analysis of Controlled Dangerous Substances (CDS) in test materials. While this method provides general guidance and structure to the analytical process, due to the unpredictability of real-world samples, method variations may occur. In such cases, the deviations must be recorded as per Agency standards, either as a Minor or Major deviation (Defined in DOM17).

2. Background

2.1. To establish the best practices for operations within the Forensic Chemistry Unit and to ensure conformance to the requirements of the Department of Forensic Sciences (DFS), the accreditation standards under ISO/IEC 17025:2017, and any supplemental standards.

3. Safety

3.1. Read Material Safety Data Sheets (SDS) to determine the safety hazards for chemicals and reagents used in the standard operating procedures.

3.2. Wear personal protective equipment (e.g., lab coat, gloves, mask, eye protection), when carrying out standard operating procedures.

3.3. Note: Do not add water to acid, only add acid to water.

4. Materials Required

4.1. Sample Extraction Materials

4.1.1. Chemicals

4.1.1.1. Chemicals should be of sufficient quality to ensure minimal interference using a mass spectrometric (MS) technique.

4.1.1.2. 18MΩ Water (may be lab-generated)

4.1.1.3. 10% solution of hydrochloric acid (HCl, *ca*. 1.2 Molar)

4.1.1.4. Concentrated Sodium Hydroxide (NaOH, *ca.* 20 Molar)

4.1.1.5. Solvent Extraction Solution(s), *e.g*.,

4.1.1.6. Chloroform

4.1.1.7. Acetonitrile

4.1.1.8. Methanol

4.1.1.9. Water

4.1.2. Equipment

4.1.2.1. Centrifuge

4.1.2.2. Gas Chromatographer/Mass Spectrometer (GC-MS) (10µL syringe suggested)

4.1.3. Consumables

4.1.3.1. Centrifuge tubes

4.1.3.2. Pipette tips

4.1.3.3. Auto sampler vials and caps

4.2. Small beaker (*e.g.,* 10mL)

4.3. Glass rod for mixing/crushing

4.4. GC-MS Materials

4.4.1. Chemicals

4.4.1.1. Methanol (MS grade, for needle washing)

4.4.1.2. Acetonitrile (MS grade, for needle washing)

4.4.1.3. Helium gas (99.999%, for carrier gas)

4.4.2. Equipment

4.4.2.1. Agilent 5975MS or Agilent 7890B GC-MS, or equivalent

4.4.2.2. Gerstel Autosampler (if used), or equivalent

4.4.2.3. Consumables

4.4.2.4. 2 mL vials and caps (9mm PTFE caps, or equivalent)

4.4.2.5. 250µL insert sleeves

4.4.2.6. GC-MS Column, *e.g.,* Restek Rx5ms, HP-5, HP-35, DB-5 or equivalent.

4.5. Liquid Chromatography Mass Spectrometry (LC-MS) Materials (LC-MS not currently in use)

4.5.1. Chemicals

4.5.1.1. Mobile Phase A – 10% Methanol (*aq)*

4.5.1.1.1. Step 1: Add 5mL: concentrated formic acid to 1L volumetric flask

4.5.1.1.2. Step 2: Add 100 mL of methanol (LC-MS grade) to same flask

4.5.1.1.3. Step 3: Add about 500 mL 18MΩ water to same flask, mix

4.5.1.1.4. Step 4: Dilute to mark with 18MΩ Water

4.5.1.2. Mobile Phase B – Acetonitrile with 5mM Formic Acid

4.5.1.2.1. Step 1: Add 5 mL of concentrated formic acid to 1L volumetric flask

4.5.1.2.2. Step 2: Dilute to mark with acetonitrile (LC-MS grade)

4.5.2. Equipment

4.5.2.1. Agilent 6460 LC-MS, ABSciex 4000, or equivalent

4.5.2.2. Consumables

4.5.2.3. 2mL vials and caps

4.5.2.4. 250µL insert sleeves

4.5.2.5. LC-MS column, Phenomenex Synergi 2.5 5µm Polar RP, or equivalent

4.6. Fourier Transform Infrared Spectroscopy (FT-IR) Materials

4.6.1. Chemicals

4.6.1.1. Reagent Grade Methanol (MeOH) or better

4.6.2. Equipment

4.6.2.1. Nicolet iS50 FT-IR with built-in ATR, or equivalent

4.6.2.2. Perkin Elmer Spectrum Two with built-in ATR, or equivalent

4.6.2.3. Polystyrene, Procaine, and Caffeine Standards

5. Standards and Controls

5.1. Standards are available from authorized vendors that manufacture ISO Guide 34 accredited products.

6. Performance Check

6.1. The GC-MS, LC-MS and Fourier Transform InfraRed Spectroscopy (FT-IR) must be brought into good operating order (for GC-MS, ion peak widths of ca. 0.6 and background oxygen and water concentration of less than 10%). If parameter cannot be maintained, consult with Unit Manager.

6.2. Analysis of a blank control shall be performed prior to sample analysis

6.2.1. If a background for a particular ion is present, then a combination of cleaning followed by blank samples to verify reduction in background is suggested

6.2.2. If a clean background cannot be successfully obtained, consult with Unit Manager.

7. Procedures

7.1. Analytical Scheme

7.1.1. The flowchart below provides guidance for analysis of unknown substances, *etc*. for presence of controlled substances, *e.g.,* synthetic cannabimimetic agents. Sample size or other circumstance may require rearrangement or modification of one or more steps, but changes must be documented in the case file.

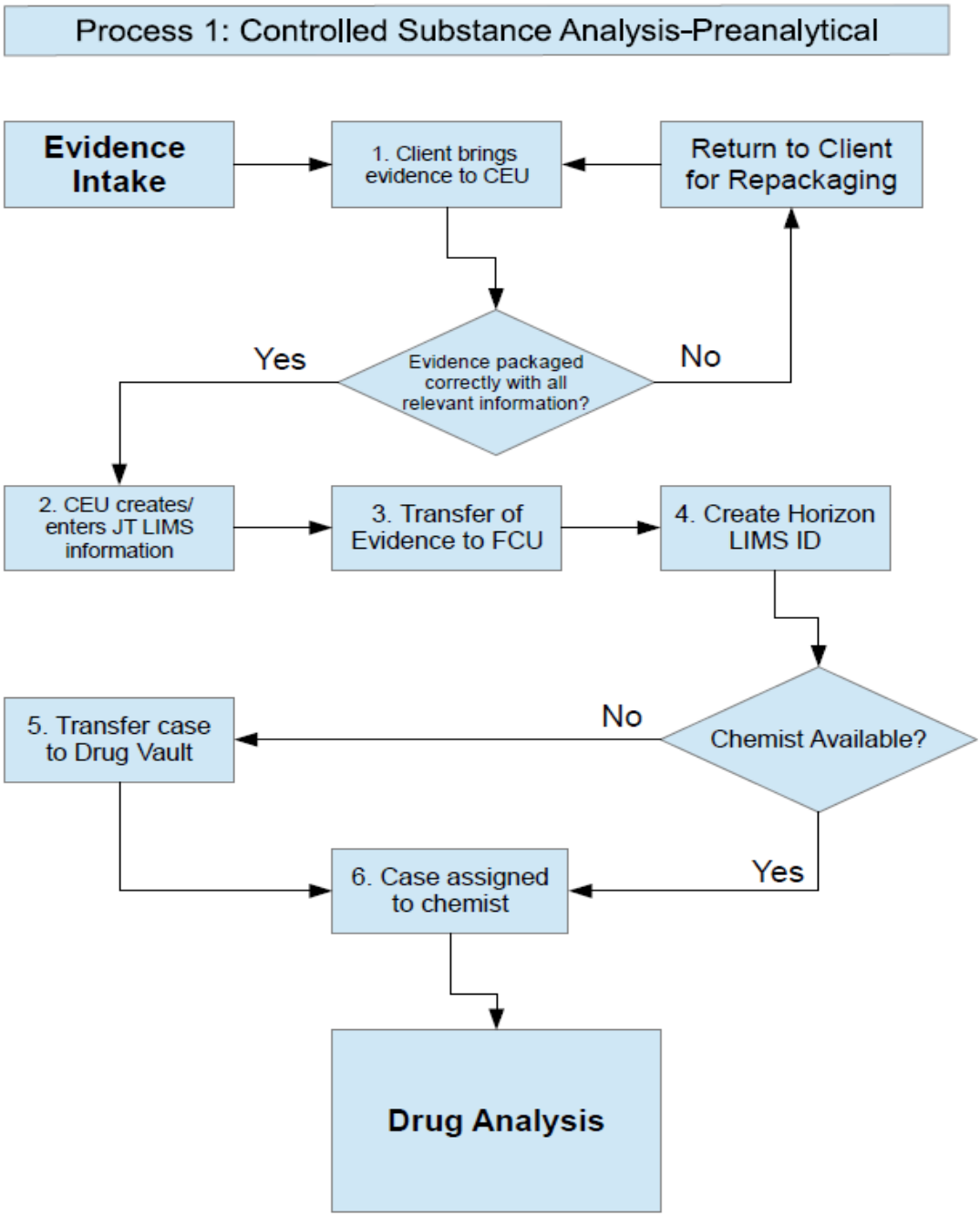

Figure 1a. Process map / flow chart for the analysis of potential or known Controlled Dangerous Substances (CDS).

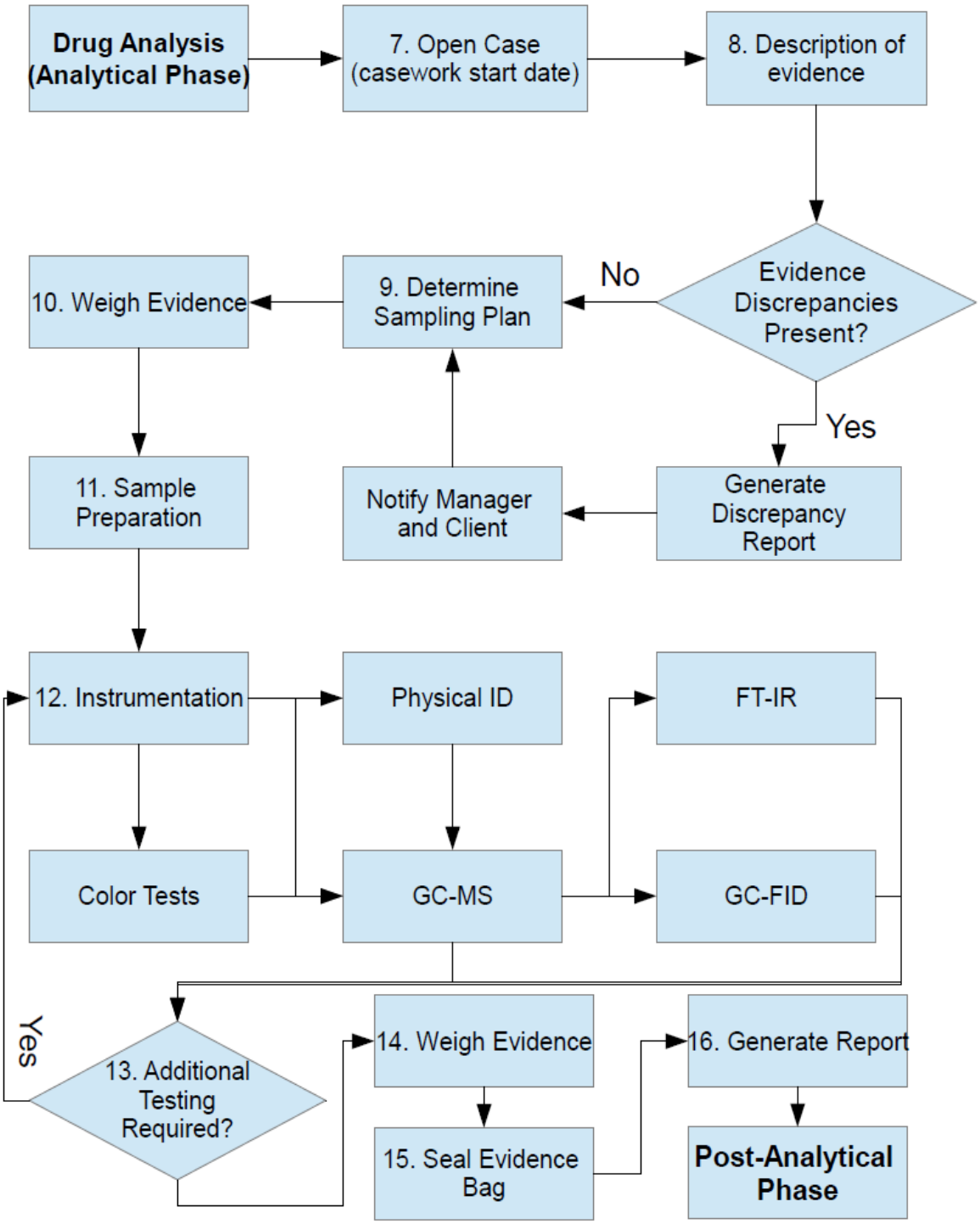

Figure 1b. Process map / flow chart for the analysis of potential or known Controlled Dangerous Substances (CDS).

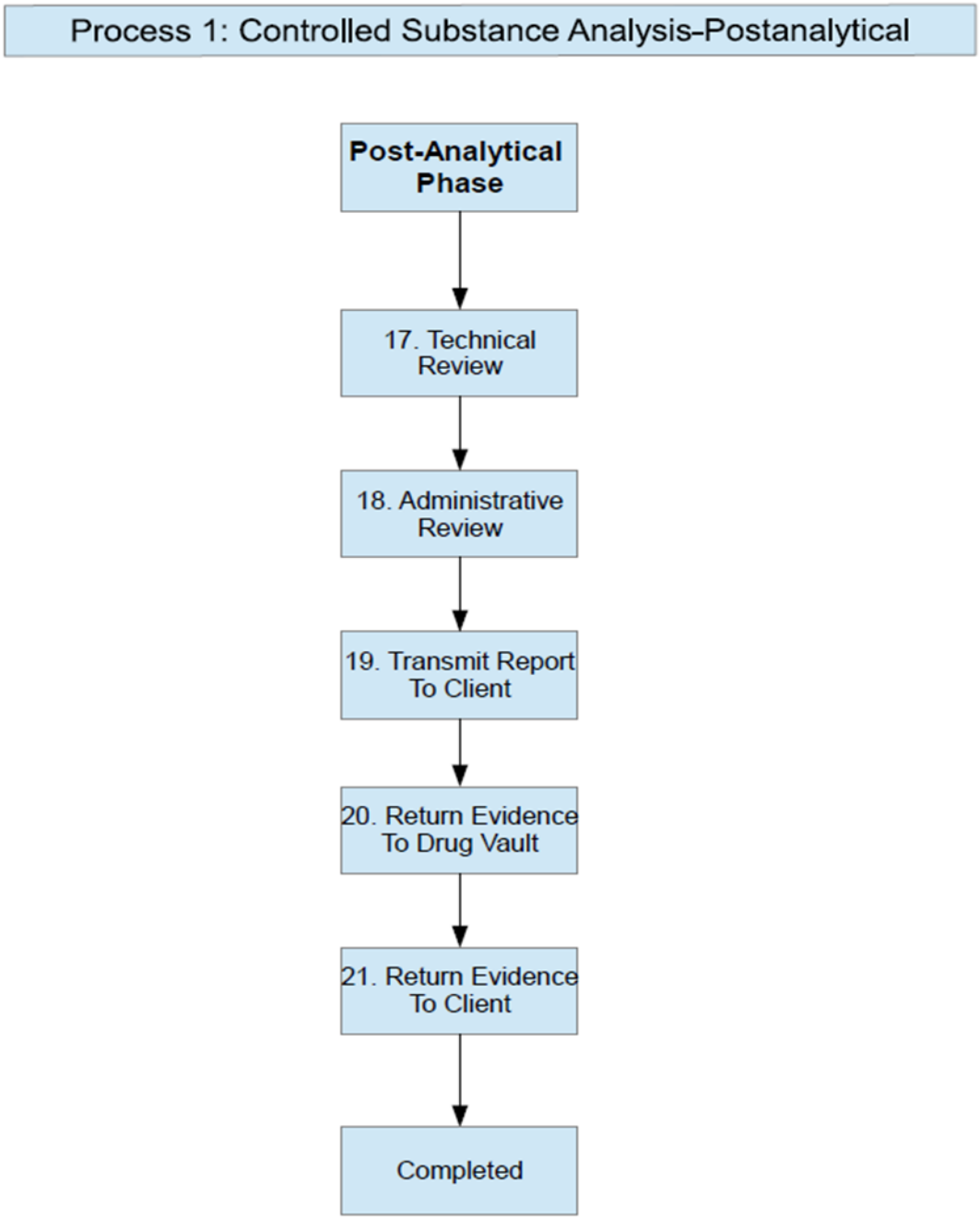

Figure 1c. Process map / flow chart for the analysis of potential or known Controlled Dangerous Substances (CDS).

7.1.2. A definitive structural identification technique such as GC/MS is required to be used on all substances where the identities will be reported.

7.1.3. If the sample is still an unknown or other confirmation is needed, the analyst should use any instrumental techniques available (or combinations thereof), such as LC-MS, to arrive at a sound analytical conclusion about the identities of the sample. (See ASTM E2329-17 for combination parameters).

7.1.4. This analysis method may be applied to the identification of non-controlled substances, if requested. The applicability of the method to the analysis of the chemical of interest will be evaluated prior to use, and identification based on a chemically pure standard as per standard procedure for drug analysis.

7.2. Weighing Practices

7.2.1. Weights for powders and other plant materials will be taken prior to sampling.

7.2.2. Net Weights

7.2.2.1. Samples without packaging (besides the heat-sealed evidence bag) shall be recorded as net weight.

7.2.2.2. Weights of capsules, cigars, and cigarettes with or without filter tips are considered net weights.

7.2.2.3. Residues will be considered a net weight.

7.2.2.4. Net weights shall be obtained and reported whenever possible by subtracting the weight of empty packaging from the gross weight (including packaging).

7.2.3. Gross Weights

7.2.3.1. Any weight that includes packaging is a gross weight, unless otherwise noted.

7.2.3.2. Analytical, top-loading or high-capacity electronic balances are acceptable for routine casework. The balance used will be recorded in the case notes.

7.2.3.3. Weights will be recorded in the analytical notes as they are displayed on the balance.

7.2.4. If the estimated uncertainty is equal or larger than the weight, a more accurate balance shall be used or the substance shall be reported as a residue, whichever is appropriate.

7.2.5. When available balances allow for it, net weights of multiple specimens in the same case item shall be measured and recorded individually. Measuring and recording the net weight of multiple specimens at the same time shall be avoided whenever it is possible to do so.

7.2.6. When multiple balances are used to record net weights of specimens within one case item, the sum of the weights recorded with each balance shall be reported separately.

7.3. Weighing Practices for Administrative Sampling Plan

7.3.1. Simple Possession

7.3.1.1. The net weight of the one specimen analyzed will be obtained and designated as a net weight (NW) in the case notes (if possible). The gross weight of the remaining (unanalyzed) specimens including innermost packaging will be obtained and designated as such in the case notes.

7.3.1.2. In cases where the container weight is clearly much greater than the sample weight, the net weight (without packaging) of the material will be obtained and reported appropriately.

7.4. Weighing Practices for Hypergeometric Sampling Plan

7.4.1. Initial Submissions

7.4.1.1. The net weight of each specimen requiring analysis will be obtained and designated as such in the case notes.

7.4.1.2. The gross weight of the remaining specimens including innermost packaging will be obtained and designated as such in the case notes.

7.4.1.3. Both the net weight of the analyzed specimens and the gross weight of the remaining specimens will be reported.

7.4.2. Resubmissions

7.4.2.1. Only the weights of the additional samples tested will be obtained and reported.

7.5. Extractions Guidance

7.5.1. Sample extraction

7.5.1.1. Obtain a representative sample of test material.

7.5.1.2. Homogenize material, as appropriate.

7.5.1.3. Determine appropriate extraction method and follow as applicable.

7.5.2. Solvent extraction (organic extraction) - as appropriate

7.5.2.1. Add appropriate amount of organic solvent (*e.g.,* acetonitrile, methanol, chloroform, hexane, *etc*.) to the sample.

7.5.2.2. Transfer solvent containing sample to a centrifuge filter vial (or to a syringe filter).

7.5.2.3. Centrifuge and/or filter the vial, and remove top layer for analysis.

7.5.2.4. Obtain the supernatant, as appropriate.

7.5.3. Solvent extraction (Acid Extract) - as appropriate

7.5.3.1. Add 50 to 100mg of test material to a centrifuge tube.

7.5.3.2. Add 1mL acetonitrile to the centrifuge tube.

7.5.3.3. Add 150µL (about 3 drops) 10% HCl to the centrifuge tube, mix thoroughly.

7.5.3.4. Add 1mL solvent extraction to the centrifuge tube, mix thoroughly.

7.5.3.5. Centrifuge tube, remove bottom organic layer (Acid Extract), keep top aqueous layer in centrifuge tube.

7.5.4. Solvent extraction (Base Component) - as appropriate

7.5.4.1. Add 100µL (about 2 drops) concentrated NaOH to the centrifuge tube containing the aqueous layer.

7.5.4.2. Add 1mL solvent extraction to the centrifuge tube, mix thoroughly.

7.5.4.3. Centrifuge tube, remove bottom organic layer (Base Extract).

7.5.5. As appropriate, combine the two extracts (acid and base) for analysis.

7.5.6. Note: In some cases, it may be decided to derivatize the test compound to enhance sensitivity. If this is performed, the analyst must record a description of the derivatization process chosen within the case packet.

7.6. Color Tests

7.6.1. Color tests will be performed on select materials as a presumptive test and must be accompanied with additional testing.

7.6.2. Each set of color tests will be accompanied with a corresponding blank.

7.6.3. When running multiple of the same color test at once, only one blank for the set is necessary.

7.6.4. The results of a color test will be indicated by writing down the observed color change (along with “Positive”) or lack thereof (along with “Negative”).

7.7. Physical Identification

7.7.1. Physical identification will be performed on pharmaceutical preparations when possible.

7.7.2. Physical identification is an examination of the evidence and comparison to a known credible reference standard.

7.7.3. Sources that may be used for this purpose are one of the following:

7.7.3.1. Drug Identification Bible

7.7.3.2. Pillbox

7.7.3.3. Physician’s Desk Reference

7.7.3.4. Ident-A-Drug

7.7.3.5. Manufacturer’s Reference

7.7.3.6. Poison Control Center

7.7.4. A secondary reference can be used to provide pictures but must be accompanied by an acceptable source.

7.7.5. If items which may be physically identified are submitted, and the sampling plan would indicate that they would not be tested, the items that would not be tested will instead be physically identified.

7.7.6. Partial pill fragments may be physically identified if they are mixed with intact pills and their physical characteristics are consistent with the intact pills.

7.8. Instrumentation

7.8.1. Gas Chromatographer/Mass Spectrometer (GC-MS)

7.8.1.1. GC-MS is two tests in tandem and is a full confirmatory test. (Scientific Working Group for the Analysis of Seized Drugs, "SWGDRUG," Category A + B tests; ASTM E2329-17 Standard Practice for Identification of Seized Drugs, Table 1)

7.8.1.2. Samples are dissolved in an appropriate organic solvent and injected on the GC-MS in a validated method.

7.8.1.3. Each substance that is to be confirmed using this technique must be compared against a standard using the same method and instrument and must fit the following criteria:

7.8.1.3.1. The retention time of the sample (the analyte of interest) must match the retention time of the corresponding standard within 2% (the sample retention time must be ±2% of the retention time of the standard or within 0.05 minutes, whichever is higher).

7.8.1.3.2. The standard must be run within the same weekly sequence as the sample to be usable for confirmation.

7.8.1.3.3. Blank runs must be run before each sample and each standard.

7.8.1.3.4. Blanks must be clear of any visible peaks.

7.8.1.3.5. The mass spectrum of the sample must match a known reference such as a mass spectral library, either in-house or provided by a known source. The mass spectrum should also match that of the standard. The determination of a match shall be made by the analyst as per their training and experience in identifying unknowns. Note: The detection is at the sensitivity of the samples to be run.

7.8.1.4. Acceptance criteria for GC-MS are provided here:

	Acceptance Criteria	Detail
GC PARAMETERS (GC-MS)	**Retention Time**	Retention time of analyte peak must match within 2% of standard or within 0.05 minutes, whichever is higher.
	S/N* Cut Off	Analyte TIC** must be more than three (3) times greater than noise, based as peak TIC of acetonitrile blank.
	Peak width resolution	Analyte peak must be base peak resolved, as evaluated by the analyst.

*S/N = Signal-to-Noise Ratio

**TIC = Total Ion Chromatogram

	Acceptance Criteria	Detail
MS PARAMETERS (GC-MS)	**Peak number**	At least two (preferred three) characteristic peaks.
	Relative peak intensity	Analyte peak relative intensity shall match their reference standards by no more than 15%.
	Peak height	Peak signal intensity must be at least 500 a.u.**

*m/z = mass-to-charge ratio

**a.u. = arbitrary units

7.8.1.5. Substances that are confirmed will be reported as "*substance name* detected".

7.8.1.6. Substances that are found but are not controlled or confirmed will be reported as "*substance name* noted".

7.8.1.7. Substances that are found but are not confirmed due to a lack of comparable standard, bad mass spectral match, or any other reason will be reported as “possible *substance name*” and cannot be used for conclusions.

7.8.1.8. Control Chart Maintenance

As appropriate, the significant parameters appropriate for the identification of cocaine (or other substance used for quality control) shall be recorded in the laboratory control chart for GC-MS. Critical pieces of information include peak retention time.

7.8.2. **Gas Chromatography Flame Ionization Detector (GC-FID)**

7.8.2.1. Unlike GC-MS, GC-FID counts as only one test and is not confirmatory by itself. (SWGDRUG Category B test, ASTM E2329-17)

7.8.2.2. Samples are dissolved in an appropriate organic solvent and injected on the GC-FID in a validated method.

7.8.3. Each substance that is to be confirmed using this technique must be compared against a standard using the same method and instrument and must fit the following criteria:

7.8.3.1. The retention time of the sample (the analyte of interest) must match the retention time of the corresponding standard within 2% (the sample retention time must be ±2% of the retention time of the standard or within 0.05 minutes, whichever is higher).

7.8.3.2. The standard must be run within the same week as the sample to be usable for matching.

7.8.3.3. Blank runs must be run before each sample and each standard.

7.8.3.4. Blanks must be clear of any visible peaks. The evaluation shall be made by the analyst as per their training and experience. Note: The detection is at the sensitivity of the samples to be run.

7.8.4. Substances that are confirmed using this technique will be reported as “*substance name* detected”.

7.8.5. If a GC-FID run is performed, but no substances are matched against it, the run will be reported as “No matching peaks detected”.

7.8.6. GC-FID Parameters

	Acceptance Criteria	Detail
GC-FID PARAMETERS	**Retention Time**	Retention time of analyte peak must match within 2% of standard or within 0.05 minutes, whichever is higher.
	S/N* Cut Off	Analyte TIC must be more than three (3) times greater than noise.
	Peak width resolution	Analyte peak must be base peak resolved, as evaluated by the analyst.

*S/N = Signal-to-Noise Ratio

7.8.6.1. Control Chart Maintenance

As appropriate, the significant parameters appropriate for the identification of individual substances or mixtures of heroin, cocaine, etc. (or other substance used for quality control) shall be recorded in the laboratory control chart for GC-FID. Critical pieces of information include peak retention time.

7.8.7. Fourier Transform Infrared Spectroscopy (FTIR)

7.8.7.1. FTIR is a confirmatory test when coupled to a Category B or lower test. (SWGDRUG Category A test, ASTM E2329-17).

7.8.7.2. A background spectrum must first be taken of the empty Attenuated Total Reflectance (ATR) diamond.

7.8.7.3. A blank scan must then be taken of the empty ATR to ensure the ATR diamond is clear.

7.8.7.4. Solid samples are directly placed on the ATR portion of the FTIR or first extracted with an organic solvent.

7.8.7.5. Liquid samples must be evaporated and dried before they are analyzed using FTIR.

7.8.7.6. Control Chart Maintenance

7.8.7.6.1. As appropriate, the significant parameters appropriate for the identification of procaine (or other substance used for quality control) shall be recorded in the laboratory control chart for FT-IR. Critical pieces of information include peak width and position of characteristic peaks.

7.8.7.7. FTIR Parameters

	Acceptance Criteria	Detail
FTIR PARAMETERS	Major peaks match relative height	As evaluated by the analyst; ideally within 20%.
	Position of major peaks	As evaluated by the analyst; ideally within $5cm^{-1}$.
	Peak width match	As evaluated by the analyst; ideally within 20%.

8. Sampling

8.1. Sampling procedure is outlined in *FCS02 – SOP for General Laboratory Procedures for FCU.*

9. Calculations

9.1. All calculations will be recorded in the case notes with appropriate uncertainty provided.

10. Uncertainty of Measurement

10.1. When quantitative results are obtained, and the significance of the value may impact the report, the uncertainty of measurement must be determined.

10.2. If a full uncertainty study has not yet been performed and calculated, the vendor-provided uncertainty in measurement shall be provided, pending an on-site validation.

11. Limitations

11.1. See specific method SOP for limitations on analytical processes.

11.2. Limitations must be clearly conveyed within the laboratory report.

12. Documentation

12.1. FCU Examination Worksheets

12.2. FCU Report of Results

13. References

13.1. Forensic Chemistry Unit Quality Assurance Manual, (Current revision).

13.2. DFS Departmental Operations Manuals, (Current revision).

13.3. Scientific Working Group for the Analysis of Seized Drugs (SWGDRUG), Recommendations, (Current revision).

13.4. Controlled Substances Procedures Manual, Department of Forensic Sciences, Virginia (DFS Document 221-D100, Rev. 18).

13.5. Diamond, F. Identification of Synthetic Cannabinoids in Herbal Incense Blends by GC/MS (NMS Labs, written for Agilent, Application Compendium, 2012).

13.6. ASTM E2329-17, Standard Practice for Identification of Seized Drugs.